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OXIDATION, VOLATILIZATION, AND REDISTRIBUTION OF MOLYBDENUM FROM TZM ALLOY IN AIR

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**Oxidation, Volatilization, and Redistribution
of Molybdenum from TZM Alloy in Air**

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ABSTRACT

The excellent high temperature strength and thermal conductivity of molybdenum-base alloys provide attractive features for components in advanced magnetic and inertial fusion devices. Refractory metal alloys react readily with oxygen and other gases. Oxidized molybdenum in turn is susceptible to losses from volatile molybdenum trioxide species, $(\text{MoO}_3)_m$, in air and the hydroxide, $\text{MoO}_2(\text{OH})_2$, formed from water vapor. Transport of radioactivity by the volatilization, migration, and re-deposition of these volatile species during a potential accident involving a loss of vacuum or inert environment represents a safety issue. In this report we present experimental results on the oxidation, volatilization and re-deposition of molybdenum from TZM in flowing air between 400 and 800°C. These results are compared with calculations obtained from a vaporization mass transfer model using chemical thermodynamic data for vapor pressures of $\text{MoO}_3(\text{g})$ over pure solid MoO_3 and an expression for the vapor pressures of $\text{MoO}_2(\text{OH})_2$ from the literature. Calculations correlate well with experimental data. The volatilization process is dominated by MoO_3 above 550°C and by $\text{MoO}_2(\text{OH})_2$, formed from the small ingress of water vapor, at temperatures below 550°C. Partial saturation of gaseous species of (MoO_3) near specimen surfaces accounts for observed reductions in volatilization rates at lower flow rates at 700°C. We have thus demonstrated predictive capabilities of the model to account for volatilization as influenced by temperature, humidity (vapor content), and flow rate.

We obtained oxidation rates (mm/h) as indicated by the recession into the base metal. These rates which accounted for the concurrent processes of oxidation and volatilization showed trends similar to other refractory metals, namely, niobium and tantalum.

Deposition of MoO_3 downstream at lower temperatures was calculated with a model using saturation ratios of $(\text{MoO}_3)_m$ within segmented regions. Calculated locations of peak distributions and maximums within the temperature gradients generally correlate reasonably well with experimental measurements. Scanning electron microscopy showed that deposits collected in a final filter consisted of small agglomerated particles. We might expect such nucleation, growth and agglomeration of particles to result from the supersaturation of the $(\text{MoO}_3)_m$ upon cooling during transport. Hydroxide molecules also decompose back into $\text{MoO}_3(\text{s})$ and water vapor at low temperatures. These latter two processes and increased surface areas due to extensive crystal growth from $(\text{MoO}_3)_m$ are plausible explanations for differences in peak height and distribution predictions between the model and experiments..

The oxidation-driven mobilization data, along with activation calculations determining radioactive isotope inventories, were used to determine airborne dose rates. These calculations showed that site boundary doses from TZM alloy would be one to two orders of magnitude lower than tungsten at comparable temperatures.

SUMMARY

In this report we present the results of experimental and modeling approaches used to define the oxidation, oxidation-driven vaporization, and re-condensation behavior of TZM alloy tested in air environments between 400 and 800°C. Experimental procedures are presented in Section 2. This section describes the test system used to expose specimens at various temperatures and flow rates in an air environment. Included are descriptions of the system and procedures used to collect, quantify, and examine the volatilized and re-deposited oxide products.

Experimental results are presented in Section 3. This includes the integration of mass change and inductively coupled plasma-atomic emission spectroscopy (ICP-AES) results to account for the concurrent processes of oxygen pick-up and oxide volatilization. These integrations provide oxidation rates (mm/h), the relative amount of the oxide that is volatilized, and the mobilization rate of molybdenum ($\text{g/m}^2\text{-h}$). Features of the oxidized specimens based on physical appearance, optical metallography, scanning electron microscopy (SEM), and x-ray diffraction (XRD) are given in Section 3.2. Illustrations of re-deposited oxides are shown in Section 3.3. Detailed descriptions of deposited oxide products on various system components, the chemical analyses procedures, and ICP-AES results providing mass flux calculations and distribution information are given in Appendices A, B, and C, respectively.

Experimental results are discussed in Section 4. Oxidation rates, mechanisms and the relative amount volatilized at various temperatures are compared to the available information in the literature in Sections 4.1 and 4.2. A description of our vaporization mass transfer model including the procedure to handle partial saturation of volatilizing oxide species near specimen surfaces is given in Section 4.3. The treatment for partial saturation helped to explain flow rates effects. In this section we also present equilibrium vapor pressures for both volatilizing polymers of $(\text{MoO}_3)_m$ and $\text{MoO}_2(\text{OH})_2$ as a function of temperature and various water vapor concentrations. The combined results from these two species, with $\text{MoO}_2(\text{OH})_2$ dominating at low temperatures and $(\text{MoO}_3)_m$ dominating at high temperatures, provide a very good correlation with experimental measurements. Our model predicting the re-deposition from $(\text{MoO}_3)_m$ vapors is described and compared with experimental molybdenum profiles in Section 4.4. The model provides good correlation with the locations of peak deposition for tests performed at and above 600°C. Plausible reasons for differences between predicted and measured peak heights and subsequent deposition profiles include: 1) nucleation, growth, agglomeration, and transport of particles due to supersaturation of $(\text{MoO}_3)_m$ upon cooling, 2) extra surface areas for re-deposition resulting from crystal development, 3) the role of $\text{MoO}_2(\text{OH})_2$ in the mobilization process at lower temperatures. A comparative study showing mobility-based doses derived for TZM alloy and tungsten is given in Section 4.5. This comparison shows that doses from the activated products of TZM alloy are one to two orders lower than tungsten for similar accident conditions of a conceptual fusion device.

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1.0 INTRODUCTION

The potential of high temperature strength has motivated much interest in the oxidation behavior of refractory metals. Many of these studies have been oriented toward extremely high temperatures, e.g., up to 1700°C, often under vacuum or environments with low oxygen activities [1,2,3,4]. Studies [1,5,6] on molybdenum at lower temperatures in high levels of oxygen report: 1) parabolic rate law at 250 to 450°C, 2) linear behavior above 400°C, 3) a role of MoO_2 and other oxides (MoO_Z), where $2 < Z < 3$, between 450 to 650°C. At temperatures above 650°C, oxidation rates are largely influenced by the high rates of the vaporization of the various polymers of MoO_3 . Water vapor will also react with $\text{MoO}_3(\text{s})$ to form a volatile hydroxide. Vapor pressures of this $\text{MoO}_2(\text{OH})_2$ species have been documented by Speiser and St. Pierre [6]. We performed this study to explore conditions more typical to future fusion devices and to demonstrate predictive capabilities of a vaporization mass transport model. The model had provided excellent mass transport predictions for the highly volatile species produced in the tungsten-steam system.[7]

2.0 EXPERIMENTAL PROCEDURES

We tested specimens of TZM alloy between 400 and 800°C in air flowing at a rate of 1000 sccm. Flow rates were varied from 10 sccm to 2500 sccm for tests at 700°C. Gas velocities for these flow rates at 700°C, the pressure at the INEEL, and the test chamber cross section ranged from 0.033 to 8.3 meters per minute. The specimens were prepared from a 22-mm diameter bar made by vacuum arc casting and then hot extrusion. The composition was Mo – 99.25 wt%, Ti - 0.50 wt%, Zr - 0.102 wt%, C - 0.018 wt%, Si – 0.0034 wt%, Fe – 0.001 wt%, Ni - 0.001 wt%, O - 0.0009 wt%, and N – 0.0005 wt%. Disc-shaped specimens of either 1.75- or 3.5-mm thickness were sectioned from the bar. Surfaces were polished to a 600-grit finish.

The specimens were tested in a dual furnace arrangement as shown in Figure 1. The test system consisted of three regions, the reaction furnace, a transition zone, and a furnace in which deposition occurred. The dimensions of the quartz components in the system were as follows: reaction chamber (4.0 cm I.D. x 36 cm long), the transition zone (1.5 cm I.D. x 13 cm long), and the deposition tube (1.5 cm I.D. x 90cm long). Air from a compressed cylinder flowed through a column of Drierite and was preheated to 400°C before passage into the reaction furnace containing the specimen. Upon exiting the reaction furnace the gas stream then passed through a transition zone and a three-zone furnace with a prescribed temperature gradient. Oxidation products transported from the specimen were re-deposited in these components downstream at temperatures between 800 and 25°C. The quartz collection tube in the three-zone furnace was lined with sections of 1-in., or 3-in., long quartz tube. These were used to determine the distribution to the re-deposited molybdenum oxide products along the temperature gradient. A final filter of quartz wool was used to ensure the complete capture of products at the end of the collection tube.

Figure 1. Schematic of test system to study the mobilization/deposition of MoO_3 from TZM molybdenum alloy in flowing air.

Specimens were weighed before and after testing to obtain mass change. The quartz components were visually inspected following the tests. Descriptions of the oxide products deposited on the components are given in Appendix A. The quartz components were then separately processed as described by the procedures in Appendix B. This involved cleaning the components with 2.9 molar $\text{NH}_4(\text{OH})$ solution to remove the molybdenum oxide products. The solutions were heated to drive off NH_3 and then acidified with nitric acid and diluted to 100 ml or 250 ml samples. The diluted solutions were analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). We examined oxidized specimens with optical metallography, scanning electron microscopy (SEM), and x-ray diffraction (XRD).

3.0 EXPERIMENTAL RESULTS

3.1 OXIDATION AND MOBILIZATION RATES. The test matrix with temperatures, times, and flow rates, along with mass changes and total amounts of molybdenum transported as measured by ICP-AES are given in Table 1. Oxidation rates and molybdenum mass flux rates can be obtained from the data in Table 1. However, mass changes need to be corrected to allow for concurrent oxygen pick-up and losses by molybdenum oxide volatilization. For these calculations we assume that the oxidation process essentially produces molybdenum trioxide by the reaction: $\text{Mo} + (3/2) \text{O}_2 \rightarrow \text{MoO}_3$. The total mass of molybdenum (M_t^{Mo}) reacted can then be determined by the relationship expressed in Eq.(1):

$$(M_t^{\text{Mo}}) = (M_v^{\text{Mo}}) + (96/48)[\Delta m + (M_v^{\text{Mo}})], \quad (1)$$

where Δm is the mass change and (M_v^{Mo}) is the amount of volatilized molybdenum as measured by ICP-AES. The results of these calculations are given in Table 2. We used these values to determine recession rates (mm/h) and the fraction of oxidized molybdenum that volatilized. Recession considered as the depth of alloy converted to oxide was calculated using 10.2 g/cm^3 as the density of molybdenum and initial surface area. We used metallography to measure recession rates (mm/h) for a limited number of specimens. Average mass flux values were obtained from the sum of the ICP-AES measurements for all system components for a given test, the initial surface area of the specimen, and the test duration. The detailed calculations from the ICP-AES measurements are reported in Appendix C. The calculations in Appendix C show the average mass flux from the specimen, molybdenum distributions, and temperature profiles. Essentially there was no indications of molybdenum mobilization at 400°C . We use a flux value, which we refer to as the Engineering Maximum Flux Value (EMFV), at this temperature. It is determined from the ICP-AES detection limit from a blank solution. The flux values at 500°C are orders of magnitude higher than the detection limit and more than ten times greater than the uncertainty based on ICP-AES limitations. A summary of the measurements and calculations refer to above are shown in Table 2.

Table 1. Test matrix with mass changes and ICP-AES measurements.

Specimen	Temp., °C	Flow rate, sccm	Time, h	Initial area,cm ²	Initial weight, g	Final weight, g	Mass change, g	ICP-AES total, g
TZM14	400	1000	24	10.345	13.6641	13.6684	0.0043	1.5E-6(a)
TZM15	500	1000	24	10.369	13.8827	13.94643	0.06373	1.02E-4
TZM1	500	1000	24	9.101	6.6733	6.7429	0.0696	5.97E-5
TZM16	550	1000	24	10.402	14.0636	14.4353	0.3717	9.04E-4
TZM11	602	1000	8	10.235	13.15934	13.35724	0.19790	3.25E-3
TZM7	599	1000	24	9.136	6.88303	7.65951	0.7765	3.56E-3
TZM8	650	1000	8	9.123	6.8263	7.3607	0.5344	2.51E-2
TZM4	699	10	2	8.994	6.1262	6.1887	0.0625	4.50E-3
TZM17	700	10	8	10.311	13.4955	13.7485	0.2530	1.91E-2
TZM5	699	50	2	9.111	6.7467	6.7988	0.0521	4.55E-3
TZM18	700	50	8	10.326	13.5565	13.7589	0.2024	5.75E-2
TZM10	699	100	2	9.112	6.71857	6.74414	0.02557	3.08E-2
TZM6	700	500	2	9.075	6.4623	6.4560	-0.0063	6.49E-2
TZM12	700	1000	2	10.343	13.67683	13.67773	0.0009	5.43E-2
TZM9	700	2500	2	9.069	6.52977	6.52907	-0.0007	5.67E-2
TZM3	750	1000	1	9.056	6.48254	6.1071	-0.3754	3.46E-1
TZM2	800	1000	1	9.150	6.9709	3.6501	-1.9056	2.22E+0

(a) The value reported for 400°C is a maximum as established by the detection limit from ICP-AES.

Table 2. Oxidation and mass flux rates determined for molybdenum oxidation tests.

Specimen	Temp °C	Flow rate, sccm	Time h	Total Mo reacted, g	Calculated recession rate,mm/h	Measured recession rate,mm/h	Fraction of reacted Mo volatilized	Measured mass flux g/(m ² -h)	Predicted mass flux g/(m ² -h)
TZM14	400	1000	24	0.0086	3.40E-5		1.74E-4	6.04E-5(a)	8.22E-5
TZM15	500	1000	24	0.1277	5.03E-4		8.00E-4	4.10E-3	2.97E-3
TZM1	500	1000	24	0.1393	6.25E-4		4.30E-4	2.73E-3	2.97E-3
TZM16	550	1000	24	0.7457	2.93E-3		1.21E-3	3.62E-2	2.28E-2
TZM11	602	1000	8	0.4053	4.85E-3		8.02E-3	3.97E-1	2.97E-1
TZM7	599	1000	24	1.5628	6.99E-3	6.24E-3	2.28E-3	1.62E-1	2.97E-1
TZM8	650	1000	8	1.1434	1.54E-2		2.19E-2	3.43E+0	4.17E+0
TZM4	699	10	2	0.1384	7.55E-3		3.25E-2	2.50E+0	7.06E-5
TZM17	700	10	8	0.5631	6.69E-3		3.40E-2	2.32E+0	7.06E-5
TZM5	699	50	2	0.1178	6.33E-3		3.86E-2	2.50E+0	4.60E-1
TZM18	700	50	8	0.5745	6.85E-3		9.95E-2	6.95E+0	4.60E-1
TZM10	699	100	2	0.1435	7.72E-3	1.12E-2	2.15E-1	1.69E+1	2.81E+0
TZM6	700	500	2	0.1820	9.83E-3		3.57E-1	3.57E+1	2.79E+1
TZM12	700	1000	2	0.1645	7.80E-3		3.30E-1	2.62E+1	4.93E+1
TZM9	700	2500	2	0.1687	9.12E-3		3.36E-1	3.13E+1	8.67E+1
TZM3	750	1000	1	0.2873	3.11E-2	5.46E-2	1.20E+0	3.82E+2	4.75E+2
TZM2	800	1000	1	2.8435	3.05E-1	3.85E-1	7.80E-1	2.42E+3	3.79E+3

(a) The value reported for 400°C is a maximum as established by the detection limit from ICP-AES.

3.2 FEATURES OF OXIDIZED SPECIMENS.

3.2.1 SPECIMEN APPEARANCE. The oxidized specimens of the TZM alloy were photographed at low magnification. Photographs in Figure 2 show specimens tested at various temperatures. Increasing amounts of residual oxide are apparent with increasing temperatures and times for up to 650°C. Above 700°C the amount of residual oxide is notably less due to oxide evaporation. The samples exposed at 750 and 800°C have only a thin darker oxide layer. The oxide on the specimen exposed at 800°C shows evidence of having been molten. It flowed down the surfaces of the specimen and formed a glob at the bottom. Figure 3 shows photographs of specimens tested at 700°C under various flow conditions. Data in Table 2 show that there were comparable amounts of molybdenum metal oxidized for all flow conditions, however, significantly greater amounts were volatilized with the higher flow rates. This is reflected in the appearance of the specimens in Figure 3. The specimens with the lower flow rates have crystals developed on the surfaces. Volatilizing molybdenum trioxide species apparently were not swept away but instead re-deposited on preferentially oriented seed crystals located on the specimen surfaces. The specimens with higher flow rates had smoother surfaces and evidence of a darker underlying oxide showing that more of the oxide was volatilized and transported from the specimens in agreement with the ICP-AES measurements.

Oxidized Specimens: Air Flow (1 liter/minute)

Figure 2. Specimens of TZM alloy oxidized at various temperatures and times in air.

Effect of Flow Rate on Specimens Oxidized at 700°C

Figure 3. Specimens of TZM alloy tested in air at 700°C with different flow rates.

3.2.2 MICROSTRUCTURAL OBSERVATIONS OF OXIDIZED SPECIMENS. Oxides formed on specimens were examined with the aid of optical metallography (OM), SEM and XRD. Metallographic cross sections showed both thicknesses of residual oxide layers and profiles of oxide formations on the specimens. A specimen exposed for 24 hours at 500°C possessed a 0.02-mm thick oxide layer with some irregularly distributed crystals extending to a height of 0.065 mm. A SEM image of surface oxides formed under such conditions is shown in Figure 4. Thicker oxide scales with multiple layers were apparent for higher temperature tests. A dense oxide layer with intrusions into the metal matrix and cusps-like formations on the outer oxide layer is shown for the 24-hour 600°C test in Figure 5. The outer regions of the oxide also show numerous cracks caused by the growth stresses in the oxide. This suggests that the surface area available for evaporation could be significantly higher than the initial geometric area of the specimen. However, the specimen tested for 8 hours at 600°C not showing such exaggerated oxide growth had a higher mass flux rate (see Table 2). This indicates that instead of the cracks within the oxide contributing to mobilization, the cusp-like formation may influence the flow pattern and reduce volatilization. The inner oxide layer and the extent of external oxide crystal growth depended upon the flow rate for 700°C tests. Figure 6 shows that the oxides existed primarily as externally protruding crystals on the specimens with a flow rate of 10 sccm. The specimen with a flow rate of 100 sccm had a more continuous 0.06-mm thick layer but still some external crystal growth (see Figure 7). Figure 8 shows only a 0.04-mm thick oxide layer with no evidence of external crystal growth for the specimen tested with a flow rate of 1000 sccm. This

correlates with the higher mass flux shown for TZM12 in Table 2. The thickness of the residual oxide layer was significantly less for tests at 700°C and higher. OM and SEM in Figure 9 show that the oxide layer for the 750°C is about 0.013-mm thick. OM provides evidence of crystal structure in the oxide layer while SEM provides better resolution of the porosity and layers within the oxide. A cross section of the very thin oxide layer (less than 0.01 mm) and glob-like formation near the bottom of the specimen tested at 800°C is shown in Figure 10. The thin residual oxide layers for the 750°C and 800°C tests agree with information in Table 2 showing that most of the oxide that formed on the specimens evaporated.

Figure 4. Oxides formed on the surface of specimen oxidized at 500°C for 24 hours.

(a)

(b)

Figure 5. Cross section of specimen oxidized in air 24 hours at 600°C. (a) Cusp-like oxide scale showing numerous cracks from growth stresses. (b) Layer at oxide to metal interface.

Figure 6. Oxide crystals on specimen oxidized at 700°C for two hours with 10 sccm flow.

Figure 7. Oxide formed on specimen oxidized at 700°C for two hours with 100 sccm flow.

Figure 8. Oxide scale formed on specimen at 700°C after two hours with 1000 sccm flow.

(a)

(b)

Figure 9. Residual oxide scale on specimen oxidized at 750°C for one hour with a flow rate of 1000 sccm. (a) optical micrograph (magnification: 400X). (b) SEM at 1000X.

Figure 10. Thin oxide layer and glob-like formation from molten oxide on specimen oxidized at 800°C for one hour with 1000 sccm flow rate.

3.2.3 PHASE AND CHEMISTRY DETERMINATIONS. Semi-quantitative analyses by energy dispersive x-ray spectroscopy (EDS) were obtained with the SEM from both cross sections and surfaces of oxidized specimens. In nearly all cases, i.e., including analyses near the oxide to metal interface and at outer regions of the oxide the molybdenum to oxygen ratio most closely indicated MoO_2 . In addition, many analyses within the base metal indicated high oxygen level, e.g. up to 25 at%. The oxygen content in the base metal does not agree with phase diagram information showing no solubility of oxygen in molybdenum. XRD analyses from the surfaces of specimens tested at 550°C (T2M16) and 600°C (T2M7) indicated only MoO_3 as shown by the spectrum in Figure 11. We could not detect any MoO_2 . Its presence, however, as a thin oxide layer at the oxide to metal interface as suggested by some authors cannot be discounted since XRD will not detect minor phases. The confirmation of MoO_3 by XRD and oxygen indications from the base metal causes us to question the validity of the EDS analyses. High molybdenum concentrations produce an excitation peak, which is located very near the oxygen peak. Perhaps the EDS software cannot appropriately handle background subtractions or other analytical corrections associated with the excitation peak.

Figure 11. XRD spectrum from specimen oxidized at 600°C for 24 hours showing only the presence of MoO₃.

3.3 FEATURES OF RE-DEPOSITED OXIDE.

The test components were examined after the test and the type, color and location of re-deposited oxide were recorded. Detailed records for each test are given in Appendix A. Photographs were taken of the collection tube for some of the tests. Some of these tests showed a start, or heavy concentration, of crystal growth at about 12 inches along the collection tube where temperature decreased to around 550 to 600°C. This region corresponded to the highest concentration of re-deposited molybdenum as determined by ICP-AES. Downstream from this region showing distinctive crystal growth there was often another type of deposit having either a smoky or sometime bluish hue. Sometimes a fairly heavy blue deposit collected at the front end of the quartz wool filter. Figure 12 shows the re-deposited oxides and molybdenum distribution for a test run at 700°C for two hours with a flow rate of 500 sccm. The SEM micrograph in Figure 12 shows that the product collected in the quartz wool consists of agglomerations of very small particles. Figure 13 shows an extremely heavy crystalline deposit for the 800°C tests. The deposit bridges and appears to nearly fill the collection tube at this location. Descriptions of deposits, molybdenum deposition profiles, and temperature profiles for all of the other tests are given in Appendices A and C.

Figure 12. Oxide deposition products from specimen tested in air at 700°C for two hours with a flow rate of 500 sccm.

Figure 13. Yellow crystalline deposit and white smoky deposit formed in the collection tube for a test run at 800°C for one hour with a flow rate of 1000 sccm.

4.0 DISCUSSION

4.1 OXIDATION RATE OF TZM ALLOY. We have plotted the recession rates, fractions of “reacted” metal volatilized, and mass flux rates from Table 2 with respect to reciprocal temperature. The plot for the recession rates is shown in Figure 14. The plot shows a good correlation between rates obtained by the (Δm + ICP-AES) and metallographic methods. The plot shows a maximum at 650°C (the rate at 650°C is 2 to 3 times higher than at 600 and 700°C), and then sharply increasing rates from 700 to 800°C. This trend in oxidation rate is very similar to those observed for the other refractory metals of niobium[2] and tantalum[3]. The irregularities are attributed to various non-stoichiometric phases other than Nb_2O_5 or Ta_2O_5 . Speiser and St. Pierre[6] reported that they sometimes observed a thin MoO_2 layer, or an external MoO_3 layer with a thin sub-layer of MoO_2 or other oxides during the oxidation of molybdenum in air between 450 to 770°C. The other oxides were reported as being non-stoichiometric, i.e., MoO_z with “z” varying between 2 and 3. The similar characteristics of the refractory metals and the observations above suggest the maximum at 650°C in Figure 14 is real and likely caused by different types of oxide. Although we have little data to show kinetic behavior, the two tests at 600°C indicate linear or even slightly accelerating behavior (Table 2).

4.2 VOLATILIZATION PROCESS FROM OXIDIZED TZM ALLOY. The plot of calculated fraction of “oxidized” molybdenum volatilized in Figure 15 show a marked increase above 650°C. Generally 20 to 33 percent of the oxide that formed volatilized at 700°C except for the tests with the lowest flow rates of 10 and 50 sccm. Although all 700°C tests formed similar amounts of oxides (Table 2), only about one-tenth as much of the oxide volatilized at the lower flow rates. Our model subsequently presented shows this is the result of partial saturation of the gas phase with the MoO_3 , effectively reducing the volatilization at low flow rates. Most of the oxide was volatilized at 750 and 800°C. This corresponds with our metallographic observations of only a thin (3 to 10 μm) oxide on these specimens. The high rate of volatilization is apparently linked to the increase in oxidation rate for these two temperatures as shown in Figure 14. Our data agree with that of Gulbransen, et al.[1], where all of the oxygen reacted at 800°C in 76 torr oxygen and formed volatile oxides. The specimen tested at 800°C showed evidence of a molten layer. The sides were smooth and parallel indicative of molten oxide flowing down the specimen. A thicker region at the bottom of the specimen contained

Figure 14. Oxidation rates of TZM alloy as shown the rate of metal reacted (mm/h).

Figure 15. The fraction the MoO_3 volatilized plotted with respect to temperature.

some oxides as revealed in the metallographic cross section. This agrees with temperatures reported for the melting points of MoO_3 (795°C) and a MoO_3 - MoO_2 eutectic (778°C)[6]. The higher vapor pressure above the melting point would increase volatilization rates, which in turn would increase oxidation rates by removing the oxide barrier.

Insight into the mechanisms governing oxidation and volatilization processes of molybdenum metal at temperatures from 827 to 1127°C is provided by Olander and Schofill[4]. They believe that very thin coatings of MoO_2 exist even at high temperatures where rapid evaporation occurs by the overall reaction: $\text{Mo(s)} + 3/2 \text{O}_2(\text{g}) = 1/m (\text{MoO}_3)_m(\text{g})$. Although the influence of oxygen adsorption on the oxidation behavior of refractory metals has been widely reported,

Olander, et al.[4] relate the dissociative adsorption of oxygen as the controlling mechanism via the reaction: $O(ads) + MoO_2 \rightarrow MoO_3(ads)$. The available active sites facilitating the adsorption of oxygen in turn depend upon the anion vacancy concentration in the n-type semiconductor structure of molybdenum dioxide. The above theory may help explain some of our data at lower temperatures. The volatilization rate for the 24-h test of $0.162 \text{ g/(m}^2\text{-h)}$ was less than the rate of $0.397 \text{ g/(m}^2\text{-h)}$ for the 8-h test at 600°C . Perhaps the stoichiometry of the oxide changed as the oxide scale grew with time and influenced the number of active sites. It is also possible that as the oxide thickened the flow pattern around the specimen changed decreasing the volatilization rate.

4.3 MODEL OF VOLATILIZATION RATES. We used a vaporization mass transfer model to predict the mobilization of molybdenum from the specimens tested in air. The model based on the experimental geometry, thermal hydraulic conditions and vapor pressures of the volatile trioxides, $(MoO_3)_m$, and hydroxide, $MoO_2(OH)_2$, was used to predict the mobilization mass flux of molybdenum, J , as expressed by Eq.(2).

$$J = k(p^{Mo} - p^{bulk})/RT \quad (2)$$

where

k = mass transfer coefficient (m/s),

p^{Mo} = partial pressure of volatile molybdenum species at surface of the specimen,

p^{bulk} = partial pressure of volatile molybdenum species in bulk steam flow.

Since the flow in the experiment is laminar, the mass transfer coefficient is given by [8]:

$$k = 1.86[Re Sc(l/d)]^{1/3} D/d \quad (3)$$

D is the diffusion coefficient of the volatile molybdenum species in air based on classic Chapman-Eskong theory [8] and Re is the Reynolds number, Sc is the Schmidt number, and l and d are the length and diameter of the furnace. Surface to bulk pressures of the volatile molybdenum species are corrected for the partial saturation of the species in the bulk flow by Eq. (4), where Q is the volumetric flow rate of air (m^3/s).

$$(p^{bulk}/p^{Mo}) = [1 - \exp(-hA/Q)] \quad (4)$$

The partial pressure of $(MoO_3)_m$ is determined as an effective value for the various polymeric forms of (MoO_3) existing at different temperatures over pure solid or molten MoO_3 . Vapor pressures for the various polymer species were obtained from the HSC thermochemical computer code[9]. Correlations [10] for the effective average polymer number (m) in $(MoO_3)_m$ were also determined. The primary species within our temperatures of interest are $(MoO_3)_3$, $(MoO_3)_4$, and $(MoO_3)_5$. Average polymer numbers above solid (MoO_3) are given by Eq. (5).

$$m = 6.785 - 0.013193T + 1.432 \times 10^{-5}T^2 - 4.4235 \times 10^{-9}T^3 \quad (5)$$

The total vapor pressures of these average effective polymers over the solid phase up to the melting point as derived from the HSC thermochemical code are shown by Eq. (6).

$$\log_{10}P(\text{Torr}) = 8,599.81/T - 186.495 + 63.1654 \log_{10}T - 0.0078447T - 4,154,834/T^2 \quad (6)$$

Vapor pressures of the hydroxide formed by the reaction: $\text{MoO}_{3(s)} + \text{H}_2\text{O}_{(g)} = \text{MoO}_2(\text{OH})_{2(g)}$, have been experimentally determined by Speiser and St. Pierre.[6] They reported an equilibrium constant, K_m , for the reaction as expressed by $K_m = P_{\text{hydroxide}}/P_{\text{water}}$, to be given by Eq. (7).

$$\log K_m = -7731/T + 5.45 \quad (\text{for } T = 873 \text{ to } 963 \text{ K}) \quad (7)$$

We obtained vapor pressures for the hydroxide species by using the relationship for K_m , the saturation pressure for water vapor in ambient temperature air of 17.55 Torr (2340 Pa), and the relative humidity. We used a parametric analysis to show vapor pressures of the hydroxide species at various relative humidities. The humidity at the INEEL is typically around 40 percent. Although we dried the air prior to introduction into the test system we postulate that there was some backflow and/or leakage of ambient air into the test system. We show comparisons of the vapor pressures of $(\text{MoO}_3)_m$ in dry air and $\text{MoO}_2(\text{OH})_{2(g)}$ in environments with the various relative humidities, including a postulated test condition with a ten percent ingress, in Figure 16. The plots show that the vapor pressure of $(\text{MoO}_3)_m$ in air and $\text{MoO}_2(\text{OH})_{2(g)}$ in the postulated experimental condition are about equivalent at 550°C. In higher relative humidities, e.g., 80 to 100 percent, the temperature at which the vapor pressures of the two different species become equivalent would increase to about 650°C.

Figure 16. Vapor pressures of molybdenum trioxide and hydroxide species at various relative humidities and temperatures.

We calculated contributions to the molybdenum mass flux from both the trioxide and hydroxide species using Eq. (2) through Eq. (7). Comparisons of predicted total mass flux of molybdenum with experimental measurements for the various times, temperatures, and flow rates are given in Table 2. The predictions for tests with a flow rate of one liter per minute are plotted along with the experimental data in Figure 17. The calculations show that below 550°C contributions from $\text{MoO}_2(\text{OH})_{2(g)}$ dominate the volatilization process, whereas $(\text{MoO}_3)_m$ is the major contributor above this temperature. The model incorporating both species agrees quite well with experimentally derived mass flux measurements over the entire temperature range. Simnad and Spilners [5] showed a very similar trend for molybdenum metal oxidized between 500 and 770°C including an inflection in volatilization rate at 600°C. They reported activation energies of 53.0 kcal/mole and 89.6 kcal/mole below and above 650°C, respectively. We obtained activation energies of 41.4 kcal/mole between 400 and 500°C and 85.3 kcal/mole between 600 and 800°C, respectively. We believe this supports the proposal that the hydroxide and trioxide species, respectively, are the active mechanisms in the two different temperature regions.

The trend of lower mass flux with lower flow rates at 700°C (shown in Table 2) is supported by our predictions using Eq. (3). The measured values, however, are still somewhat higher than predicted values for the lowest flow rates. Protruding crystals were observed growing from specimens tested in the low flow rates as evident in Figure 3 and Figure 6. We would expect environments with higher partial saturation to be more conducive to grow such crystals from preferential sites. Some of these crystals were quite needle, or ribbon-like, and would easily break off. In fact, ribbon and needle-like crystals were observed in the reaction tubes for 700°C tests with 10 and 50 sccm flow rates and molybdenum measurements were higher compared to the other components (see Appendices A and C). This supports the suggestion that the high measurements for mobilized material compared to predicted values result from spalled crystals.

Figure 17. Mass flux of molybdenum showing experimental and predictions from the $(\text{MoO}_3)_m$ and $\text{MoO}_2(\text{OH})_{2(g)}$ vapor species at various temperatures.

4.4 MODEL FOR RE-DEPOSITION OF MOBILIZED MOLYBDENUM.

In this section, we present a simple condensation model that predicts the condensation profiles in the downstream components in the experiments. The $(\text{MoO}_3)_m$ vapors are assumed to be the primary species vaporized from the sample particularly at 600°C and higher as shown by the preceding section. For a given flow rate in the experiment, a concentration of $(\text{MoO}_3)_m$ in the gas phase is established by Eq. (8):

$$C_{\text{gas}} = (\Gamma \cdot A) / Q \quad (8)$$

where,

$$\begin{aligned} C_{\text{gas}} &= \text{concentration of } (\text{MoO}_3)_m \text{ in gas (kg/m}^3\text{),} \\ \Gamma &= \text{measured mass flux from surface (kg/m}^2\text{-s),} \\ A &= \text{initial surface area of sample (m}^2\text{), and} \\ Q &= \text{volumetric flow rate of air (m}^3\text{/s).} \end{aligned}$$

As a packet of gas travels down the tube, it cools and the $(\text{MoO}_3)_m$ becomes supersaturated. The saturation ratio expressed by Eq. (9) is that defined by Friedlander[11]:

$$S = C_{\text{gas}}(R \cdot T) / (p_{\text{sat}}(T) \cdot \text{MW}) \quad (9)$$

where,

$$\begin{aligned} S &= \text{saturation ratio,} \\ \text{MW} &= \text{molecular weight of } (\text{MoO}_3)_m \text{ (kg/kgmole),} \\ R &= \text{gas constant (Pa-m}^3\text{/kgmole-K),} \\ T &= \text{temperature (K), and} \\ p_{\text{sat}}(T) &= \text{vapor pressure of } (\text{MoO}_3)_m \text{ at temperature T} \end{aligned}$$

The vapor pressure of $(\text{MoO}_3)_m$ was obtained from the HSC thermochemical computer code[9].

Condensation to the wall will occur when the concentration in the gas exceeds the equilibrium vapor concentration at the given temperature. The rate of condensation given by Eq. (10) in that presented in Ref. [8]:

$$J_{\text{cond}} = k (C_{\text{gas}} - C_{\text{equ}}) \quad (10)$$

where,

$$\begin{aligned} J_{\text{cond}} &= \text{condensation mass flux (kg/m}^2\text{-s),} \\ k &= \text{mass transfer coefficient (m/s), and} \\ C_{\text{equ}} &= \text{equilibrium vapor concentration (kg/m}^3\text{).} \end{aligned}$$

Using the ideal gas law to relate the equilibrium vapor concentration to the vapor pressure and the definition of supersaturation ratio, this relationship can be written as Eq. (11).

$$J_{\text{cond}} = k A_s \text{MW } p_{\text{sat}}(T) (S-1) / (R \cdot T) \quad (11)$$

The mass transfer coefficient expressed as k in Eq. (12) is given by Bird[8]:

$$k = 1.86 * (D/d) * [\text{Re} * \text{Sc} (L/d)]^{0.333} \quad (12)$$

where

- Re = Reynolds number of the flow,
- Sc = Schmidt number,
- D = diffusion coefficient of $(\text{MoO}_3)_m$ in air given by the Chapman-Eskong theory which is also presented in Bird[8],
- d = diameter of tube, and
- L = length of tube from sample to location of interest along the tube.

Thus, the mass balance equations that describe the mass of $(\text{MoO}_3)_m$ condensed and in the vapor phase are:

$$dC_{\text{gas}}/dt = -J_{\text{cond}} * A_{\text{wall}}/V \quad (13)$$

$$dC_{\text{cond}}/dt = J_{\text{cond}} * A_{\text{wall}}/V \quad (14)$$

where

A_{wall}/V = surface to volume ratio of pipe equals $(4/d)$.

Converting the concentration in the gas to supersaturation yields Eq. (15)

$$dS/dt = (kA_{\text{wall}}/V) * (S-1) \quad (15)$$

Integration yields Eq. (16):

$$S(t) = 1 - (S_o - 1) \exp(-kA_{\text{wall}} * t/V) = 1 - (S_o - 1) \exp(-4kt/d), \quad (16)$$

where S_o is the initial supersaturation at the start of the collection tube. The collection tube is then discretized into an equal number of uniform cells (usually 100 over the length of the collection tube each 1 cm each). In this case S_o is the initial supersaturation as the packet of gas enters the cell and $S(t)$ is set to the final value as it exits the 1-cm long cell. This final value is given by Eq. (17):

$$S_{\text{final}} = 1 - (S_o - 1) \exp(-4k\Delta t/d) = 1 - (S_o - 1) \exp(-4kV_i/Q * d), \quad (17)$$

where

- Δt = time for the packet to transit the cell,
- V_i = volume of segment i , and
- Q = volumetric flow rate.

And the mass condensed in a given cell is given by Eq. (18):

$$[S_{\text{final}} - S_o] * p_{\text{sat}}(T) * MW / [R * T] \quad (18)$$

The final supersaturation exiting a cell is the initial supersaturation entering the next cell in the collection tube. Temperatures in the tube for each test were linearly fit (piecewise into two or three pieces in some cases to get the best fit) and then the linear fit was used in the calculations. The $(\text{MoO}_3)_m$ deposition was converted to molybdenum by multiplying by the ratio of the molecular weights. The measured and calculated depositions were converted to deposition per unit length given the limited number of measurements and the large variation in the calculated deposition along the tube.

The results for various cases of temperatures, flow rates and times as listed in Table 3 are shown in Figures 18(a) through Figure 18(h).

Table 3. Cases calculated by deposition model.

Case Number	Temperature (°C)	Flow rate of Air (slpm)	Duration (hr)
1	800	1	1
2	700	0.5	2
3	700	2.5	2
4	700	1	2
5	650	1	8
6	600	1	8
7	600	1	24
8	500	1	24

The results show that the start of condensation is very well predicted with the model for all the cases. The peak deposition is well predicted in the majority of cases (e.g., Cases 2, 3, 5 and 7) and in the worst cases overpredicts the peak deposition by between 50 and 100%. The deposit model predicts a broad tail of deposition beyond the peak deposition location. This broad tail in most cases overpredicts the observed deposition in the tube. Deviations in the peak heights and the broad tail distribution can be explained by deposition occurring at preferential macroscopic sites provided by growing crystals. For example, this is most apparent for the 800°C test that had crystals bridging the I.D. and nearly plug the tube. Deposition on these crystals would substantially deplete the $(\text{MoO}_3)_m$ concentration causing lower than predicted deposition in downstream locations. The experimental data also showed significant molybdenum deposition near the end of the tube and in the glass wool. This deposition was bluish colored and SEM showed that the deposition in the quartz wool consisted of agglomerated small particles. We might expect particle nucleation and transport upon cooling the supersaturated gas. Such aerosol formation was not considered in the model. Had nucleation been incorporated into the model, some of the condensation would have occurred onto those nucleated particles decreasing the

calculated condensation onto the tube in regions where supersaturation is high enough to support both nucleation and condensation onto both particle and walls. The role of $\text{MoO}_2(\text{OH})_{2(g)}$ was also not considered. Decomposition of this vapor at lower temperatures may have produced the oxide with the bluish color. This latter mechanism also provides an explanation for the large discrepancy for the 500°C test, i.e., Case 8. Most of the molybdenum is shown to be volatilized as $\text{MoO}_2(\text{OH})_{2(g)}$ in Section 3.3 rather than $(\text{MoO}_3)_m$. Analyses from the ICP-AES showed that most of the re-deposited oxide was in the reaction chamber as reported in Appendix C. In conclusion, given the simplicity of the model, it does a reasonable job at broadly describing deposition behavior at 600°C and higher where volatilization is dominated by $(\text{MoO}_3)_m$.

Figure 18(a). Case 1—Test at 800°C with one liter per minute flow rate for one hour.

Figure 18(b). Case 2 – Test at 700°C with 0.5 liter per minute flow for two hours.

Figure 18(c). Case 3 – Test at 700°C with 2.5 liter per minute flow for two hours.

Figure 18(d). Case 4 – Test at 700°C with one liter per minute flow for two hours.

Figure 18(e). Case 5 – Test at 650°C with one liter per minute flow rate for 8 hours.

Figure 18(f). Case 6 – Test at 600°C with one liter per minute flow for 8 hours.

Figure 18(g). Case 7 – Test at 600°C with one liter per minute flow rate for 24 hours.

Figure 18(h). Case 8 – Test at 500°C with one liter per minute flow rate for 24 hours.

4.5 MOBILITY-BASED DOSE DERIVED FROM OXIDATION DRIVEN VOLATILITY

We can use the oxidation-driven mobilization data to calculate a mobility-based dose. This dose can be used as a metric to compare with other fusion materials or with appropriate confinement factors used in a safety analysis of a conceptual fusion design.

The fractional rate of mobilization of a particular element in an alloy at a given temperature is calculated using the Eq. (19):¹²

$$\dot{MF} = \frac{\Gamma}{WP \times \rho \times \delta} \quad (19)$$

where $\dot{MF}$ is the mobilization fraction per unit time, Γ is the cumulative mass flux of the element ($\text{kg/m}^2\text{-s}$) based on the test data, WP is the weight fraction of the element in the material, ρ is the density of the material (kg/m^3), and δ is the thickness of the component (m) that is being analyzed.

The oxidation-driven mobility-based dose rate is then given by the following equation:

$$DoseRate = [RI \times DI] \times \dot{MF} \times A_{FW} \times \rho \times \delta \quad (20)$$

where,

RI = radioactive inventory of isotope per unit mass of material (Bq/kg) in the component,

DI = dose impact of isotope (Sv/Bq),

A_{FW} = surface area of first wall (m^2).

The inner summation is over all isotopes produced and the outer summation is over all of the elements in the alloy.

Thus, the dose rate is simply given by:

$$DoseRate = \frac{[RI \times DI] \times \Gamma \times A_{FW}}{WP} \quad (21)$$

For the TZM alloy, an activation calculation for the first wall based on the EVOLVE design assuming TZM as the first wall structural material was used.¹³ The dose impact of each isotope was calculated for both ground level and elevated releases using conservative meteorology (class F and 1 m/s wind speed as needed in a traditional "design basis" safety analysis), and using best estimate meteorology (class D and 4 m/s wind speed as needed in a no-evacuation assessment).¹⁴ A one-kilometer site boundary and the regulatory-accepted Pasquill-Gifford dispersion set were used in the evaluation. All results are early doses (7-day exposure) to the maximum exposed individual (at the site boundary for the ground level cases and where the plume touches the ground for the elevated releases). These data are reproduced in Table 4.

Table 4. Radioactive inventory and Dose Impact of Mo and Tc Isotopes.

Isotope	Radioactive Inventory (Ci/g)	Dose Impact D and 4 m/s Ground level (Sv/TBq)	Dose Impact D and 4 m/s Elevated (Sv/TBq)	Dose Impact F and 1 m/s Ground level (Sv/TBq)
Mo 93	2.60E-03	4.10E-05	3.72E-06	4.26E-04
Mo 93m	1.35E-02	5.43E-07	4.89E-08	5.53E-06
Mo 99	2.13E-01	6.98E-06	6.38E-07	7.23E-05
Mo101	1.83E-01	5.45E-08	7.01E-09	2.69E-07
Tc 98	1.81E-07	4.66E-05	4.27E-06	4.83E-04
Tc 99	4.88E-05	8.01E-06	7.28E-07	8.34E-05
Tc 99m	2.80E+00	1.81E-07	1.97E-08	1.73E-06
Tc101	1.83E-01	7.01E-09	8.44E-10	3.20E-08
All Mo isotope contribution to dose (Sv/g)*		5.97E-08	5.46E-09	6.16E-07
All Tc isotope contribution to dose (Sv/g)*		1.88E-08	2.05E-09	1.79E-07

* Calculated by summing the product of the radioactive inventory of an isotope and its dose impact.

The mobility-based dose rate for Mo from the TZM alloy is based on the cumulative maximum¹ method from data presented in Table 2 in Section 3.1. Mobilization of the Tc isotopes produced by activation of the Mo is assumed to follow that of the Mo at all temperatures because of the very high vapor pressure of the technetium oxides.¹⁵ The other elements in the TZM alloy did not mobilize in the experiments because of their extremely low volatility and are not included in the evaluation. The results, based on a 500 m² first wall with no radiological confinement, are shown in Table 5. They are compared graphically to the mobilization from tungsten alloy for two of the dose cases studied in Figures 19 and 20.

Table 5. Oxidation-driven Mobilization-base Dose Rates as a Function of Temperature Under Different Exposure Conditions.

Temperature (°C)	Oxidation-driven Mobilization-based Dose Rate		
	D&4 m/s Ground (Sv/hr)	D&4 m/s Elevated (Sv/hr)	F&1 m/s Ground (Sv/hr)
400	2.37E-09	2.27E-10	2.40E-08
500	1.61E-07	1.54E-08	1.63E-06
600	1.57E-05	1.50E-06	1.59E-04
650	1.35E-04	1.29E-05	1.36E-03
700	1.40E-03	1.34E-04	1.42E-02
750	1.50E-02	1.43E-03	1.52E-01
800	9.51E-02	9.10E-03	9.64E-01

The results show that the mobilization dose for TZM alloy is about two orders of magnitude lower than that for tungsten below about 600°C, and decreases to about one order of magnitude up to 800°C. Above 800°C, the Mo in the TZM that converts to molybdenum trioxide melts making measurements very difficult. The results can also be used to determine the degree of radiological confinement needed in accidents involving oxidation driven mobilization of material from the first wall. In a decay heat transient involving air ingress that reaches 750°C for five days, the oxidation driven mobilization dose would be approximately 2 Sv (see Figure 20). Thus, to meet a 10 mSv no-evacuation dose would require the radiological confinement to reduce this source term by about a factor of 200. After five days at 750°C, approximately 70 percent of the affected first wall area would have been mobilized. Transients that result in shorter times at lower temperatures would require less radiological confinement to meet the no-evacuation goal. If temperatures always remained below 500°C in a transient then this would not be a significant contribution to the off-site radiological source term in an accident.

¹ The concept of cumulative maximum is a conservative formulation used for safety assessments. The cumulative maximum mobility at temperature X is the maximum mobility measured at all temperatures up to and including X. Thus, if the maximum mobility measured at 600°C is higher than the value measured at 700°C, the value from the 600°C test is used in place of the value at 700°C.

Figure 19. Oxidation driven mobilization doses for W and Mo in air. The design basis for release are 500 m² surface area with no confinement.

Figure 20. Oxidation driven mobilization doses for W and Mo in air. Releases are for 500 m² surface area with no confinement and class D and 4 m/s wind speed for no-evacuation criteria.

5. CONCLUSIONS

We have observed good correlation between experimental volatilization rates and those predicted by our mass transport model. The volatilization process occurs by two different mechanisms at low and high temperatures. At low temperatures, e.g., below 550°C volatilization is dominated by $\text{MoO}_2(\text{OH})_{2(g)}$ formed from even small concentrations of water vapor. Above this temperature volatilization occurs predominately by the volatile (MoO_3) polymeric species. Our model accounted for both of these mechanisms in the two different temperature regions. It can therefore be used to accurately predict molybdenum mobilization for different climatic conditions, or ambient and humidity conditions of the air, involved in the ingress accident. The model also proved capable of accounting for flow rates which affected near-surface partial saturation of the volatilizing species and reduced mobilization.

The oxidation rate (mm/h) as indicated by recession, i.e., the amount of metal reacted, shows an increasing trend to 650°C, then a slight decrease, and then sharply higher rates due to high volatilization. This trend with a maximum near 650°C simulates that observed with other refractory metals and has been associated with different oxide types. The higher oxidation rates at higher temperature, e.g., 700 to 800°C are due to the very high volatilization rates of the molybdenum trioxide.

Our model for the re-deposition of volatilized molybdenum species of (MoO_3) accurately predicted the downstream peak-deposition locations. The model provided best results for the higher temperatures where volatilization by the various polymeric forms of (MoO_3) is dominant. Differences between predicted and experimental peak heights and subsequent deposition profiles resulted when extensive crystal growth at the peak-deposition locations provided greatly increased surface area for re-deposition to occur.

Mobility-based dose calculations derived from oxidation-driven mobilization data showed TzM alloy to have lower radiological dose impact than tungsten at comparable temperatures and conditions. Dose levels from TzM alloy were two orders of magnitude lower below 600°C and about one order of magnitude lower at 800°C.

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APPENDIX A

DESCRIPTIONS OF MOLYBDENUM OXIDE PRODUCTS DEPOSITED IN QUARTZWARE TEST COMPONENTS

Description of Oxidation Products from TZM 14

Test temperature (°C) 400 Time (h) 24 Flow rate (lpm) 1
 Specimen mass (g): Initial 13.6641 Final 13.6684 Mass gain 0.0043

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear, there is no evidence of any reaction product in test chamber.
- 2.
- 3.
- 4.

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear, there is no evidence of any reaction products in collection tube, inserts, or quartz wool filter.
- 2.
- 3.
- 4.
- 5.

Description of Oxidation Products from TZM 1

Test temperature (°C) 500 Time (h) 24 Flow rate (lpm) 1
 Specimen mass (g): Initial 6.6733 Final 6.7429 Mass gain 0.0696

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Nothing apparent in the reaction chamber

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear to 4.5 inches	
2. Light smoky deposit (insert I2)	4.7 to 7
3. Lighter smoky deposit (I3 and I4)	7 to 15.5
4. Slight color (on I5 and I6)	15.5 to 16.5
5. Nothing apparent (I7 to I9)	16.5 to 25
6. Very slight coloring (I9 and I11)	25 to 31.5
7. Nothing apparent on quartz wool	

Description of Oxidation Products from TZM 15

Test temperature (°C) 500 Time (h) 24 Flow rate (lpm) 1
 Specimen mass (g): Initial 13.8827 Final 13.94643 Mass gain 0.06373

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear, there is no evidence of any reaction product in test chamber.
- 2.
- 3.
- 4.

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear, there is no evidence of any reaction products in collection tube, inserts, or quartz wool filter.
- 2.
- 3.
- 4.
- 5.
- 6.

Description of Oxidation Products from TZM 16

Test temperature (°C) 550 Time (h) 24 Flow rate (lpm) 1
 Specimen mass (g): Initial 14.0636 Final 14.4353 Mass gain 0.3717

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Nothing apparent

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear for first 12.5 inches		
2. Light smoky color (E4 thru E6)	12.5 to 18.5	
3. Light smoky to light blue	18.5 to 21	
4. Very light blue deposit	21 to 22	
5. Clear	22 to 31	
6. Very light bluish hue	31 to quartz wool	

Description of Oxidation Products from TzM 11

Test temperature (°C) 600 Time (h) 8 Flow rate (lpm) 1 lpm
 Specimen mass (g): Initial 13.15934 Final 13.35724 Mass gain 0.1970

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Localized white deposit on wall	12 – 14
(This be due to the close positioning of the specimen to the wall of the reaction tube or to a localized cold spot in the wall of the reaction tube.)	

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Start of light white deposit	12.5
2. Light, white deposit	12.5 – 15.5
3. Heavier white, smokey deposit	15.5 – 27.5
4. Clearer region, less deposit	27.5 - 34
5. Blue-grey deposit in front of quartz wool	34 - 35
6. Back of quartz wool is still white	35 – 36

Description of Oxidation Products from TZM 7

Test temperature (°C) 600 Time (h) 24 Flow rate (lpm) 1
 Specimen mass (g): Initial 6.88303 Final 7.65951 Mass gain 0.7765

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (°C)
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1. Nothing apparent in the reaction chamber.
- 2.
- 3.
- 4.

Deposition chamber

Description of oxidation products	Location (inches from left)	Temp. (°C)
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1. Clear, nothing present	0 to 10
2. Sporadic small crystals with small particles	10 to 11
3. Smoky deposit, i.e., small particles	11 to 13
4. White, to smoky, to light blue deposits	13 to 21.5
5. Clear	21.5 to 34
6. Quartz wool is blue in front and white in back	

Description of Oxidation Products from TZM 8

Test temperature (°C) 650 Time (h) 8 Flow rate (lpm) 1
 Specimen mass (g): Initial 6.8263 Final 7.3607 Mass gain 0.5344

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. clear, nothing in the reaction chamber.
- 2.
- 3.
- 4.

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear through first three inserts.	0 to 10.25	
2. Small crystals	11 to 12	
3. Heavy, white smoky deposit.	12 to 14.5	
4. Medium white smoky deposit.	14.5 to 16.5	
5. Clearing (no evidence of deposit)	17	
6. Quartz wool is very blue in front, clear (white) in back.		

Description of Oxidation Products from TZM 4

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 0.010
 Specimen mass (g): Initial 6.1262 Final 6.1887 Mass gain 0.0625

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Clear except for some silvery, flat ribbon-like crystals near outlet.		18.5 to 23
2.		
3.		
4.		

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. No apparent deposit in collection tube, inserts, or Q.W. filter.		
2.		
3.		
4.		
5.		
6.		

Description of Oxidation Products from TZM 17

Test temperature (°C) 700 Time (h) 8 Flow rate (lpm) 0.01
 Specimen mass (g): Initial 13.4955 Final 13.7485 Mass gain 0.2530

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Smoky deposit from 0 to 2.5 in., The cap has some smoky, white deposit.		
2. Silvery crystals to ribbon-like needles	2.5 to 3.5	
3. Thinly, distributed, very thin ribbon-like crystals	13.5 to 18.5	
The crystals irradient (showing green, blue, red or transparent depending upon angle of reflection).		

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Few, thin ribbon-like crystals.	6 to 12	
2. Medium to small crystals	12 to 13.25	
3. Light blue to smoky, thin deposit	13.25 to 16	
4. Regions of light smoky blue deposits	18.5 to 31.	
5. Nothing apparent on the quartz wool		

Description of Oxidation Products from TZM 5

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 0.05 lpm
 Specimen mass (g): Initial 6.7467 Final 6.7988 Mass gain 0.0521

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Small silvery needle-like crystals, widely spaced	18.5 to 19.5	
2. Very large crystals (1/8 to 1/2 in. long)	19.5 to 20.5	
3.		
4.		

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Few speck-size crystals, widely spaced.	0 to 1	
2. Mostly clear	0 to 12	
3. Small crystals, widely spaced	10.75 to 12.25	
4. Clusters of very small particles.	11.75 to 12.25	
5. Particles become smaller and disappear.	12.25 to 13	
6. Clear thereafter.	13 to 36	

Description of Oxidation Products from TZM 18

Test temperature (°C) 700 Time (h) 8 Flow rate (lpm) 0.05
 Specimen mass (g): Initial 13.5565 Final 13.7589 Mass gain 0.2024

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. There may be some slight haze on walls	9.75 to 12	
2. Distinct needle-like crystals have formed	start at @ 13.5	
3. Heavier deposit of crystals.	16 to 17.5 (end of large section)	
4. Smaller less needle-like crystals	17.5 to 22	

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Very light whitish, smoky haze	0 to 36	
2. Large ribbon-like crystals (some 0.5 in.)	9 to 11.5	
3. Heavier deposit which extends across I.D.	11.5 to 12.5	
4. Smaller crystals	12.5 to 14.5	
5. Just haze on wall	14.5 to 27	
6. Fewer, small crystals on wall	27 to 33.5	
7. Heavy collection of ribbon-like crystals in front quartz wool filter	33.5 to 34.5	
8. No indication on any color on quartz wool.		

Description of Oxidation Products from TZM 10

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 0.10
 Specimen mass (g): Initial 6.71857 Final 6.74414 Mass gain 0.02557

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Large to medium shiny, silver, flat sliver-like crystals sporadically attached:	8 to 14.	
2. Crystals on top of tube.	9 to 13	
3. Crystals on bottom of tube.	12 to 14.5	
4. Smokey deposit on top of tube.	12 to 14	

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Clear	0 to 10	
2. Silvery sliver-like crystals	10 to 13.5	
3. Smoky deposit on wall	13.5 to 17	
4. Smaller yellowish needle-like crystals	11 to 13	
5. White smoky deposit: heavy 12.5 to 16, Becomes light to clear at about 16 inches.		
6. Large silver crystals at 34 to 36 likely dislodged during transport.		
7. None to barely detectable deposit in quartz wool.		

Description of Oxidation Products from TZM 6

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 0.5
 Specimen mass (g): Initial 6.4623 Final 6.4560 Mass loss 0.0063

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear over most of the chamber.		
2. Silvery crystals on the wall	Last 2 inches	
3. The amount of crystals are less than those observed for the test with 0.05 lpm flow.		
4.		

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
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1. Very small crystals on first insert.	2 to 2.5	
2. Clear	2.5 to 10	
3. Larger silvery crystals.	10 to 12.5	
4. White deposit.	12.5 to 17	
5. Very light blue deposit	17 to 31.5	
6. Heavier blue deposit	31.5 to 34.5	
7. Quartz wool is blue in front, white in back.	34.5 to 36.	

Description of Oxidation Products from TZM 12

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 1
lpm

Specimen mass (g): Initial 13.67683 Final 13.67773 Mass gain 0.0009

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
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1. There is only a few small clear crystals at one location likely from when the specimen contacted the surface during removal.

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Formation of clear crystal start	9.75 inches	
2. Crystal become larger	9.75 to 10.75	
4. Crystals become smaller	10.75 to 12.25	
5. White to smoke color deposit	12.25 to 16.25	
6. Very light haze	16.25 to 23.5	
7. Clear region	23.5 to 29	
8. Hazy deposit increases (bluish hue)	29 to 34	
9. Quartz wool is blue in front, white in back	34 to 36	

Description of Oxidation Products from TZM 9

Test temperature (°C) 700 Time (h) 2 Flow rate (lpm) 2.5
 Specimen mass (g): Initial 6.52977 Final 6.52907 Mass loss 0.0007

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
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1. No evidence of any deposit in the reaction chamber.
- 2.
- 3.
- 4.

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
--	------------------------------------	-------------------

1. Clear, except for a few small needle-like crystals.	0 to 10.5	
2. Small crystal, heavier deposition.	10.5 to 11.5	
3. White deposit on walls.	11.5 to 17	
4. Smokey deposit	17 to 33.5	
5. Blue deposit	33.5 to 34	
6. Blue deposit in front half, white at end of quartz wool.	34 to 36	

Description of Oxidation Products from TZM 3

Test temperature (°C) 750 Time (h) 1 Flow rate (lpm) 1
 Specimen mass (g): Initial 6.48253 Final 6.1071 Mass loss 0.3754

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Silvery flat needle-like crystals (1/4-in.) at exit.	20.5 to 23	

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Silvery needle-like crystals	0 to 1	
2. Clear.	1 to 10	
3. Large greenish crystals extending across tube	10 to 11.5	
4. Med. and small crystals, heavy conc.	11 to 12.25	
5. Small crystals with crystalline particles, i.e., white smoky deposit.	12.25 to 14	
6. Heavy white deposit , crystals becoming yellowish	14 to 16	
7. White smoky deposit with greenish streaks.	16 to 25	
8. Yellow-greenish smoky deposit	25 to 31	
9. Greenish smoky deposit becomes heavier.	34.25	
10. Heavy greenish deposit in quartz wool, the end is still white.	34.25 to 36	

Description of Oxidation Products from TZM 2

Test temperature (°C) 800 Time (h) 1 Flow rate (lpm) 1
 Specimen mass (g): Initial 6.9709 Final 5.0653 Mass loss 1.9056
 Final weight includes 3.6498 g from specimen plus 1.4152 from molten oxide.

Reaction chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Two pieces of molten oxide (1.4152 g)	13.4 to 17.5	
2. Crystals changing from white, to yellow, to greenish	18.5 to 22	
3. Larger greenish crystals appear to nearly block tube at outlet.		

Deposition chamber

<u>Description of oxidation products</u>	<u>Location (inches from left)</u>	<u>Temp. (°C)</u>
1. Clear.		0 to 11
2. Few, yellow (silver) crystals		11 to 14
3. Heavy yellow, greenish crystals extend across I.D.		14 to 16
4. Heavy white smoky deposit with some crystals		16 to 20
5. Deposit becomes yellow (or light greenish) again with some crystals		20 to 34
6. Heavy conc. of yellow-greenish crystals in front of quartz wool.		34 to 35
7. Heavy yellow-greenish deposit in front of quartz wool. (The back portion to the quartz wool is still white.)		35 to 36

APPENDIX B

PROCEDURES FOR CLEANING THE MOLYBDENUM-BEARING TEST COMPONENTS AND PROCESSING THE SOLUTIONS FOR ICP-AES ANALYSES

Processing and Analytical Procedures for Molybdenum –Bearing Glassware.
for
Molybdenum Oxidation Tests
IHRG# IRC-99-740

Scope: This procedure describes chemically removing molybdenum oxidation products from quartz test components. The test components the reaction chamber, collection tube, quartz tube inserts, and quartz wool filters as described in IHRG #IRC-99-740.

The following basic procedures will be used for all components (there will be only minor differences used to handle the various components).

1. Wash the component once or twice with 2.9 Molar NH_4OH solution.
2. Rinse with nanopure water.
3. Heat solutions in covered Teflon beakers on hot plate to drive off NH_3 .
Transfer solution to 100-ml volumetric flask.
Add 5 ml of concentrated nitric acid (Normality of 15.8).
Dilute to 100 ml with nanopure water.
Analyze by ICP-AES.

The reaction chamber and 36-long collection tube will be sealed with Parafilm at on end. The 25 ml of the 2.9 M NH_4OH will then be added and the other end sealed with Parafilm. The component will then be manipulated to wash the inner surfaces. This solution will be transferred to a Teflon beaker and the process repeated with another batch of 2.9 M NH_4OH . The inside surfaces of the component will then be rinsed with nanopure water using a squirt bottle. The rinse water will be drained directly into the Teflon beaker. This rinsing process will be done at least twice. The solution collected in the Teflon beaker will be processes as indicated above.

There are two different sizes of quartz tube inserts: 1-in. long pieces and 3-in. long pieces. Molybdenum oxidation products will be cleaned from these pieces by rinsing them with of 2.9 M NH_4OH solution dispensed from a squirt bottle. Approximately 50 ml will be used to rinse each segment directly into a Teflon beaker. Each segment will then be rinsed with about 30 ml of nanopure water. The solution collected in the Teflon beaker will be processes as indicated above.

The quartz wool filter will be placed in a 50-ml plastic disposable beaker. It will be soaked in 25 ml of the 2.9 M NH_4OH solution overnight. The 25 ml of solution will be transferred to a Teflon beaker. Another 25 ml of 2.9 M NH_4OH solution will be added to the quartz wool for a second soak (approx. 10 minutes). The quartz wool will then be rinse with 2.9 M NH_4OH solution from a squirt bottle followed by a nanopure water rinse. All solutions will have been collected in the Teflon beaker which will be processes as indicated above.

APPENDIX C

**ICP-AES ANALYSES,
MASS FLUX CALCULATIONS,
MOLYBDENUM DISTRIBUTIONS,
TEMPERATURE PROFILES**

TZM14: Tested at 400°C for 24 hours (1000 sccm flow)

Test TZM14		ICP-AES measurements for 100 ml aliquots, except RXN Tube is 250 ml.		
			Mo 202.0	
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainties and 3 sigma (S.D.) for detection limits.			=====	
For Data analyzed		% SD of Cal. Stds	sigma %	3.300
on 6-21-99		SD of Blk Std	sigma prime ug/ml	0.005
TZM14	400C/24h		F1, ug/ml	-0.008
Tested 6-2-99	air		F2, ug/ml	-0.006
time	24.000		F3, ug/ml	-0.006
			F4, ug/ml	-0.008
			F5, ug/ml	-0.007
			F6, ug/ml	-0.008
area,cm2	10.34500		F7, ug/ml	-0.011
			F8,ug/ml	-0.013
			F9,ug/ml	-0.013
			F10,ug/ml	-0.012
			F11,ug/ml	-0.013
			QW,ug/ml	-0.010
Flux set by EMFV.			C.T.,ug/ml	0.000
			RXN T,ug/ml	-0.001
			subtotal, ug	-11.750
			(+/-) ug	0.228
			Blk sub, ug	-8.400
"IF" statement compares Total			(+/-) ug	3.742
with 3*SD(ug), and all "FX" & "Component"			Total, ug	-3.350
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	3.749
Total is selected if any of the				
"FX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.015
greater than 3*SD(ug/ml).			DL (3*SD), ug	1.500
		Detection Limit (EMFV)	g/m2-h	6.04E-05
		Total correct for DL (IF statement), ug		1.500
		Calculated from Total	Mass Flux, g/m2-h	-1.35E-04
		Calc. from "IF" statement	Mass Flux, g/m2-h	6.04E-05
			± g/m2-h	1.51E-04
			± %	249.91

TZM15: Tested at 500°C for 24 hours (1000 sccm flow)

Mo Oxidation Test: TZM15 ---- 500C for 24 h at 1 liter/min.					
ICP Results	202.0	Volume	Mass Mo		
sample	Mo µg/mL	sample(mL)	ug		
TZM15G1	0.010	100	1.04		
TZM15G2	0.011	100	1.1		
TZM15G3	0.012	100	1.2		
TZM15G4	0.018	100	1.82		
TZM15G5	0.013	100	1.28		
TZM15G6	0.004	100	0.38		
TZM15G7	0.001	100	0.1		
TZM15G8	0.000	100	0		
TZM15G9	0.004	100	0.35		
TZM15G10	0.003	100	0.25		
TZM15G11	0.003	100	0.34		
TZM15QW	0.013	100	1.25		
TZM15 CT	0.020	100	1.96		
TZM15 RX	0.334	250	83.475		
		Total (ug)	94.545		
		Total (mg)	0.09		
	Amount excluding RXN and Col tube C			9.11	
			Mass Mo (ug)	April,29	
	Position in.	Mass Mo ug	w Col tube redistributed	Temp., C	Temp., x 0.01
TZM15G1	2	1.04	1.3	494	4.94
TZM15G2	5	1.1	1.3	539	5.39
TZM15G3	8	1.2	1.5	508	5.08
TZM15G4	11	1.82	2.2	456	4.56
TZM15G5	14	1.28	1.6	349	3.49
TZM15G6	17	0.38	0.5	274	2.74
TZM15G7	20	0.1	0.1	238	2.38
TZM15G8	23	0	0.0	211	2.11
TZM15G9	26	0.35	0.4	187	1.87
TZM15G10	29	0.25	0.3		
TZM15G11	32	0.34	0.4		
TZM15QW	35	1.25	1.5		
TZM15 CT		1.96

TZM15: Tested at 500°C for 24 hours (1000 sccm flow)

Test TZM15		ICP-AES measurements for 100 ml aliquots, except RXN Tube is 250 ml.						
				Mo 202.0				Percent
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainties				=====			Mass Mo	in
and 3 sigma (S.D.) for detection limits.							ug	Component
For Data analyzed		% SD of Cal. Stds	sigma %	5.100				
on 7-06-99		SD of Blk Std	sigma prime ug/ml	0.004	RXN CHMBR		83.475	88.81
					CT&INSRTS		9.27	9.86
TZM15	500C/24h		G1, ug/ml	0.010	QW FLTR		1.25	1.33
Tested 6-8-99	air		G2, ug/ml	0.011				
time	24.000		G3, ug/ml	0.012	Total		93.995	
			G4, ug/ml	0.018				
			G5, ug/ml	0.013				
			G6, ug/ml	0.004				
area,cm2	10.36900		G7, ug/ml	0.001				
			G8,ug/ml	-0.006				
			G9,ug/ml	0.004				
			G10,ug/ml	0.003				
			G11,ug/ml	0.003				
			QW,ug/ml	0.013				
Flux set by EMFV.			C.T.,ug/ml	0.020				
			RXN T,ug/ml	0.334				
			subtotal, ug	93.995				
			(+/-) ug	8.524				
			Blk sub, ug	-8.400				
"IF" statement compares Total			(+/-) ug	2.993				
with 3*SD(ug), and all "IS" & "DB"			Total, ug	102.395				
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	9.034				
Total is selected if any of the								
"GX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.012				
greater than 3*SD(ug/ml).			DL (3*SD), ug	1.200				
		Detection Limit (EMFV)	g/m2-h	4.82E-05				
		Total correct for DL (IF statement), ug		102.395				
		Calculated from Total	Mass Flux, g/m2-h	4.11E-03				
		Calc. from "IF" statement	Mass Flux, g/m2-h	4.11E-03				
			± g/m2-h	3.63E-04				
			± %	8.82

TZM1: Tested at 500°C for 24 hours (1000 sccm flow)

Test TZM1	ICP-AES measurements for 100 ml aliquots, except CT (w stuck I1) is 250 ml.			
				Mo 202.0
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainties				=====
and 3 sigma (S.D.) for detection limits.				
For Data analyzed		% SD of Cal. Stds	sigma %	2.600
on 12-03-99		SD of Blk Std	sigma prime ug/ml	0.007
TZM1	500C/24h			
Tested 11-15-99	air		I2, ug/ml	0.020
time	24.000		I3, ug/ml	0.027
			I4, ug/ml	0.016
			I5, ug/ml	0.001
			I6, ug/ml	-0.003
area,cm2	9.1010		I7, ug/ml	0.007
			I8,ug/ml	0.003
			I9,ug/ml	-0.003
			I10,ug/ml	-0.007
			I11,ug/ml	-0.004
			QW,ug/ml	0.022
Flux set by EMFV.			(C.T+I1),ug/ml	0.014
			RXN T,ug/ml	0.405
			subtotal, ug	51.900
			(+/-) ug	2.127
			Blk sub, ug	-7.800
"IF" statement compares Total			(+/-) ug	5.048
with 3*SD(ug), and all "IX" & "Component"			Total, ug	59.700
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	5.477
Total is selected if any of the				
"IX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.021
greater than 3*SD(ug/ml).			DL (3*SD), ug	2.100
		Detection Limit (EMFV)	g/m2-h	9.61E-05
		Total corrected for DL (IF statement), ug		59.700
		Calculated from Total	Mass Flux, g/m2-h	2.73E-03
		Calc. from 'IF' statement	Mass Flux, g/m2-h	2.73E-03
			± g/m2-h	2.51E-04
			± %	9.18

TZM1: Tested at 500°C for 24 hours (1000 sccm flow)

TZM16: Tested at 550°C for 24 hours (1000 sccm flow)

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TZM11: Tested at 600°C for 8 hours (1000 sccm flow)

[illegible]

TZM7: Tested at 600°C for 24 hours (1000 sccm flow)

Mo Oxidation Test: TZM7 ---- 600C for 24 h at 1 liter/min.									
						Mass Mo	Percent		
ICP Results	202.0	Volume	Mass Mo			ug	in		
sample	Mo ug/mL	sample(mL)	ug				Component		
					RXN CHMBR	392.7	11.03		
TZM 7 E1	0.049	100	4.94		CT&INSRTS	2800.71	78.65		
TZM 7 E2	0.062	100	6.21		QW FLTR	367.6	10.32		
TZM 7 E3	0.061	100	6.07						
TZM 7 E4	12.730	100	1273		Total	3561.01			
TZM 7 E5	7.416	100	741.6						
TZM 7 E6	4.041	100	404.1						
TZM 7 E7	1.758	100	175.8						
TZM 7 E8	0.524	100	52.42						
TZM 7 E9	0.204	100	20.37						
TZM 7 E10	0.155	100	15.54						
TZM 7 E11	0.683	100	68.25						
TZM 7 QW	3.676	100	367.6						
TZM 7 Col Tube	0.324	100	32.41						
TZM 7 RXN	3.927	100	392.7						
		Total (ug)	3561.01						
		Total (mg)	3.56						
		Amount excluding RXN and Col tube (ug)	3135.9						
			Mass Mo (ug)	04/28/1999					
	Position	Mass Mo	w Col tube	Temp (1),					
	in.	ug	redistributed	C					
TZM 7 E1	2	4.94	5.0	593	Specimen (cm ²)				
TZM 7 E2	5	6.21	6.3	620	9.136				
TZM 7 E3	8	6.07	6.1	585					
TZM 7 E4	11	1273	1286.2	520					
TZM 7 E5	14	741.6	749.3	422					
					Rate (g/m ² -h):				
TZM 7 E6	17	404.1	408.3	354	0.16				
TZM 7 E7	20	175.8	177.6	311					
TZM 7 E8	23	52.42	53.0	274					
TZM 7 E9	26	20.37	20.6	240					
TZM 7 E10	29	15.54	15.7						
TZM 7 E11	32	68.25	69.0						
TZM 7 QW	35	367.6	371.4						
TZM 7 Col Tube		32.41							

Component	Mass Mo (ug)	Percent (%)
RXN CHMBR	392.7	11.03
CT&INSRTS	2800.71	78.65
QW FLTR	367.6	10.32

Distance (in)	Mass (ug)	Temp(1) (ug)	Temp(2) (ug)
0	0	550	550
5	0	600	600
10	1300	550	550
15	750	450	450
20	150	300	300
25	50	200	200
30	20	100	100
35	350	50	50

TZM8: Tested at 650°C for 8 hours (1000 sccm flow)

Mo Oxidation Test TZM8 ---- 650C for 8 h at 1.0 liters/minute						Mass Mo	Percent			
ICP Results	202.0	Volume	Mass Mo			ug	in			
sample	Mo µg/mL	Sample(mL)	ug				Component			
				RXN CHMBR		2757	11.01			
TZM 8 B1	0.337	100	33.7	CT&INSRTS		18595.8	74.23			
TZM 8 B2	0.439	100	43.9	QW FLTR		3698	14.76			
TZM 8 B3	7.132	100	713.2							
TZM 8 B4	104.100	100	10410	Total		25050.8				
TZM 8 B5	40.940	100	4094							
TZM 8 B6	9.321	100	932.1							
TZM 8 B7	2.387	100	238.7							
TZM 8 B8	1.641	100	164.1							
TZM 8 B9	1.447	100	144.7							
TZM 8 B10	1.942	100	194.2							
TZM 8 B11	3.922	100	392.2							
TZM 8 QW	36.980	100	3698							
TZM 8 Col Tube	12.350	100	1235							
TZM 8 RXN	27.570	100	2757							
		Total (ug)	25051							
		Total (mg)	25.1							
		Amount excluding RXN and Col tube (mg)		21059						
			Mass Mo,ug	Mass Mo,mg						
	Position	Mass Mo	w Col tube	w Col tube						
	in.	ug	redistributed	redistributed						
TZM 8 B1	2	33.7	35.7	0.0357						Aug.2,99
TZM 8 B2	5	43.9	46.5	0.0465	Specimen (cm ²)			Front	Temp.,	Temp.,
TZM 8 B3	8	713.2	755.0	0.7550	9.123			of C.T.	C	x 10
TZM 8 B4	11	10410.0	11020.5	11.0205			16	0	588	5880
TZM 8 B5	14	4094.0	4334.1	4.3341			17	1	633	6330
TZM 8 B6	17	932.1	986.8	0.9868			18	2	643	6430
TZM 8 B7	20	238.7	252.7	0.2527	Rate (g/m ² -h):		19	3	649	6490
TZM 8 B8	23	164.1	173.7	0.1737	3.43		20	4	657	6570
TZM 8 B9	26	144.7	153.2	0.1532			21	5	663	6630
TZM 8 B10	29	194.2	205.6	0.2056			22	6	660	6600
TZM 8 B11	32	392.2	415.2	0.4152			23	7	647	6470
TZM 8 QW	35	3698.0	3914.9	3.9149			24	8	640	6400
TZM 8 Col Tube		1235.0					25	9	627	6270
							26	10	615	6150
							27	11	586	5860
							28	12	550	5500
							29	13	499	4990
							30	14	466	4660
							31	15	430	4300
							32	16	397	3970
							33	17	375	3750
							34	18	361	3610
							35	19	341	3410
							36	20	326	3260
							40	24	269	2690
							44	28	228	2280
							46	30	205	2050
							48	32	166	1660
							49	33	128	1280
							50	34	84	840
							51	35	68	680
							52	36	30	300

TZM4: Tested at 700°C for 2 hours (10 sccm flow)

								Mass Mo	Percent
								ug	in
									Component
Mo Oxidation Test TZM4 ---- 700C for 2 h at 0.01 liter/min.									
						RXN CHMBR		4208	93.55
ICP Results	202.0			Volume	Mass Mo	CT&INSRTS		282.8	6.29
sample	Mo ug/mL			sample(mL)	ug	QW FLTR		7.2	0.16
						Total		4498	
TZM 4 H1	0.072			100	7.2				
TZM 4 H2	0.044			100	4.4				
TZM 4 H3	0.038			100	3.8				
TZM 4 H4	1.035			100	103.5				
TZM 4 H5	0.147			100	14.7				
TZM 4 H6	0.011			100	1.1				
TZM 4 H7	-0.004			100	-0.4				
TZM 4 H8	-0.002			100	-0.2				
TZM 4 H9	0.009			100	0.9				
TZM 4 H10	0.034			100	3.4				
TZM 4 H11	0.001			100	0.1				
TZM 4 QW	0.072			100	7.2				
TZM 4 Col Tube	1.443			100	144.3				
TZM 4 RXN	42.08			100	4208				
				Total (ug)	4498				
				Total (mg)	4.5				
				Amount excluding RXN and Col tube (ug):	145.7				
				Mass Mo (ug)	April,28				
	Position	Mass Mo	w Col tube	Temp. x 10	Temp.(1),				
	in.	ug	redistributed		C				
						Summer'99			
						Temp.(2),C			
TZM 4 H1	2	7.2	14.3	6720	672	Specimen (cm ²)	16	0	659
TZM 4 H2	5	4.4	8.8	7060	706	8.994	17	1	676
TZM 4 H3	8	3.8	7.6	6790	679		18	2	683
TZM 4 H4	11	103.5	206.0	5990	599		19	3	690
TZM 4 H5	14	14.7	29.3	4750	475		20	4	699
TZM 4 H6	17	1.1	2.2	3850	385	Rate (g/m ² -h):	21	5	708
TZM 4 H7	20	-0.4	-0.8	3270	327	2.50	22	6	707
TZM 4 H8	23	-0.2	-0.4	2820	282		23	7	696
TZM 4 H9	26	0.9	1.8				24	8	685
TZM 4 H10	29	3.4	6.8				25	9	671
TZM 4 H11	32	0.1	0.2				26	10	648
TZM 4 QW	35	7.2	14.3				27	11	609
TZM 4 Col Tube		144.3					28	12	563
							29	13	518
							30	14	476
							32	16	415
							36	20	319
							40	24	255
							44	28	220
							46	30	174
							48	32	96
							49	33	63
							50	34	39
							51	35	30
							52	36	20

Component	Mass Mo (ug)
RXN CHMBR	4208
CT&INSRTS	282.8
QW FLTR	7.2
Total	4498

TZM17: Tested at 700°C for 8 hours (10 sccm flow)

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TZM5: Tested at 700°C for 2 hours (50 sccm flow)

Mo Oxidation Test TZM5 ----- 700C for 2 h at 0.05 liters/minute						Mass Mo	Percent		
ICP Results	202.0	Volume	Mass Mo			ug	in		
sample	Mo µg/mL	Sample(mL)	ug				Component		
				RXN CHMBR		3750	82.46		
TZM 5 F1	0.179	100	17.9	CT&INSRTS		793.7	17.45		
TZM 5 F2	0.098	100	9.8	QW FLTR		4.2	0.09		
TZM 5 F3	0.118	100	11.8						
TZM 5 F4	6.181	100	618.1	Total		4547.9			
TZM 5 F5	0.368	100	36.8						
TZM 5 F6	0.153	100	15.3						
TZM 5 F7	0.009	100	0.9						
TZM 5 F8	0.009	100	0.9						
TZM 5 F9	0.079	100	7.9						
TZM 5 F10	0.104	100	10.4						
TZM 5 F11	0.000	100	0						
TZM5 QW	0.042	100	4.2						
TZM 5 FG	0.158	100	15.8						
TZM 5 Col Tube	0.481	100	48.1						
TZM 5 RXN	37.500	100	3750						
		Total (ug)	4548						
		Total (mg)	4.5						
		Amount excluding RXN and Col tube (mg)	749.8						
			Mass Mo,ug	Mass Mo,mg					
	Position	Mass Mo	w Col tube	w Col tube					
	in.	ug	redistributed	redistributed					
TZM 5 F1	2	17.9	19.0	0.0190					
TZM 5 F2	5	9.8	10.4	0.0104	Specimen (cm ²)				Summer'99
TZM 5 F3	8	11.8	12.6	0.0126	9.111				Temp., C
TZM 5 F4	11	618.1	657.8	0.6578			16	0	659
TZM 5 F5	14	36.8	39.2	0.0392			17	1	676
TZM 5 F6	17	15.3	16.3	0.0163			18	2	683
TZM 5 F7	20	0.9	1.0	0.0010	Rate (g/m ² -h):		19	3	690
TZM 5 F8	23	0.9	1.0	0.0010	2.50		20	4	699
TZM 5 F9	26	7.9	8.4	0.0084			21	5	708
TZM 5 F10	29	10.4	11.1	0.0111			22	6	707
TZM 5 F11	31	0.0	0.0	0.0000			23	7	696
TZM5 QW	35	4.2	4.5	0.0045			24	8	685
TZM 5 Col Tube		48.1					25	9	671
							26	10	648
							27	11	609
							28	12	563
							29	13	518
							30	14	476
							32	16	415
							36	20	319
							40	24	255
							44	28	220
							46	30	174
							48	32	96
							49	33	63
							50	34	39
							51	35	30
							52	36	20

TZM18: Tested at 700°C for 8 hours (50 sccm flow)

								Mass Mo	Percent
								ug	in
									Component
Mo Oxidation Test: TZM18 ---- 700C for 8 h at 0.05 liter/min.									
					RXN CHMBR			3001.0	5.22
ICP Results	202.0	Volume	Mass Mo		CT&INSRTS			53302.3	92.78
sample	Mo µg/mL	sample(mL)	ug		QW FLTR			1148.0	2.00
TZM18 H1, ug/ml	0.190	100	19.000		Total			57451.3	
TZM18 H2, ug/ml	3.359	100	335.900						
TZM18 H3, ug/ml	140.600	100	14060.000						
TZM18 H4, ug/ml	237.400	100	23740.000						
TZM18 H5, ug/ml	6.535	100	653.500						
TZM18 H6, ug/ml	0.481	100	48.100						
TZM18 H7, ug/ml	0.961	100	96.100						
TZM18 H8,ug/ml	1.547	100	154.700						
TZM18 H9,ug/ml	12.830	100	1283.000						
TZM18 H10,ug/ml	38.120	100	3812.000						
TZM18 H11,ug/ml	81.680	100	8168.000						
TZM18 QW,ug/ml	11.480	100	1148.000						
TZM18 (C.T.),ug/ml	9.320	100	932.000						
TZM18 RXN T,ug/ml	30.010	100	3001.000						
		Total (ug)	57451.300						
		Total (mg)	57.451						
		Amount excluding RXN and Col tube (ug):	53518.300						
			Mass Mo (ug)						
	Position	Mass Mo	w Col tube						
	in.	ug	redistributed						
TZM18H1	3	19	19.331				Specimen (cm ²)		
TZM18H2	5	335.900	341.750				10.326		
TZM18H3	8	14060.000	14304.849						
TZM18H4	11	23740.000	24153.423						
TZM18H5	14	653.500	664.880						
TZM18H6	17	48.100	48.938				Rate (g/m ² -h):		
TZM18H7	20	96.100	97.774				6.95E+00		
TZM18H8	23	154.700	157.394						
TZM18H9	26	1283.000	1305.343						
TZM18H10	29	3812.000	3878.384						
TZM18H11	32	8168.000	8310.242						
TZM18QW	35	1148.000	1167.992						
TZM18CT		932.000							

Detailed description: A bar chart titled 'Mo Distribution' showing the percentage of molybdenum in different components. The y-axis ranges from 0 to 100. The x-axis lists three components: RXN CHMBR, CT&INSRTS, and QW FLTR. The bars are blue. RXN CHMBR is approximately 5%, CT&INSRTS is approximately 93%, and QW FLTR is approximately 2%.

Component	Percentage (%)
RXN CHMBR	~5
CT&INSRTS	~93
QW FLTR	~2

Detailed description: A line graph titled 'Mo Collection Profile' showing micrograms of Mo collected versus distance along the collection tube in inches. The y-axis ranges from 0 to 30,000 µg. The x-axis ranges from 0 to 40 inches. A single data series named 'Mass' is plotted with square markers connected by a line. There is a small peak around 3 inches, a very large peak at approximately 11 inches reaching nearly 25,000 µg, and a smaller secondary peak around 32 inches reaching about 8,000 µg.

Distance (in.)	Micrograms of Mo collected
3	~100
5	~100
8	~14,000
11	~24,000
14	~100
17	~100
20	~100
23	~100
26	~1,000
29	~4,000
32	~8,000
35	~1,000

TZM10: Tested at 700°C for 2 hours (100 sccm flow)

Mo Oxidation Test TZM10 ---- 700C for 2 h at 0.1 liter/min.										Percent
								Mass Mo	in	
								ug	Component	
ICP Results	202.0	336.1	339.1	Volume	Mass Mo	RXN CHMBR		6940	22.53	
sample	Mo µg/m	Ti µg/mL	Zr µg/mL	sample(mL)	ug	CT&INSRTS		23118	75.04	
						QW FLTR		751.2	2.44	
TZM 10 E1	1.941	0.001	-0.002	100	194.1					
TZM 10 E2	0.424	-0.002	-0.004	100	42.4		Total	30809.2		
TZM 10 E3	21.705	0.000	0.016	100	2170.5					
TZM 10 E4	139.900	0.012	0.085	100	13990					
TZM 10 E5	3.068	-0.005	-0.006	100	306.8					
TZM 10 E6	16.260	0.000	0.003	100	1626					
TZM 10 E7	6.315	0.001	0.000	100	631.5					
TZM 10 E8	14.420	-0.001	0.000	100	1442					
TZM 10 E9	3.164	0.001	0.002	100	316.4					
TZM 10 E10	5.311	-0.003	-0.001	100	531.1					
TZM 10 E11	9.064	0.012	0.041	100	906.4					
TZM 10 QW	7.512	0.003	0.003	100	751.2					
TZM 10 CT	9.608	-0.001	0.004	100	960.75					
TZM 10 RXN	27.760	0.001	0.017	250	6940					
				Total (ug)	30809					
				Total (mg)	30.8					
				Amount excluding RXN and Col tube	22908.4					
				Mass Mo (ug)	April,28					
	Position	Mass Mo	w Col tube	Temp. x 10	Temp.,					
	in.	ug	redistributed		C					
TZM 10 E1	2	194.1	202.2	6720	672	Specimen (cm ²)			Summer'99	Temp.
TZM 10 E2	5	42.4	44.2	7060	706	9.112			Temp.(2),C	x 10
TZM 10 E3	8	2170.5	2261.5	6790	679		16	0	659	6590
TZM 10 E4	11	13990	14576.7	5990	599		17	1	676	6760
TZM 10 E5	14	306.8	319.7	4750	475		18	2	683	6830
TZM 10 E6	17	1626	1694.2	3850	385	Rate (g/m ² -h):	19	3	690	6900
TZM 10 E7	20	631.5	658.0	3270	327	16.91	20	4	699	6990
TZM 10 E8	23	1442	1502.5	2820	282		21	5	708	7080
TZM 10 E9	26	316.4	329.7				22	6	707	7070
TZM 10 E10	29	531.1	553.4				23	7	696	6960
TZM 10 E11	32	906.4	944.4				24	8	685	6850
TZM 10 QW	35	751.2	782.7				25	9	671	6710
TZM 10 CT		960.75					26	10	648	6480
							27	11	609	6090
							28	12	563	5630
							29	13	518	5180
							30	14	476	4760
							32	16	415	4150
							36	20	319	3190
							40	24	255	2550
							44	28	220	2200
							46	30	174	1740
							48	32	96	960
							49	33	63	630
							50	34	39	390
							51	35	30	300
							52	36	20	200

TZM6: Tested at 700°C for 2 hours (500 sccm flow)

Mo Oxidation Test TZM6 ---- 700C for 2 h at 0.5 liter/minute							Mass Mo	Percent	
ICP Results	202.0	Volume	Mass Mo				ug	in	
sample	Mo µg/mL	Sample(mL)	ug					Component	
				RXN CHMBR			12560	19.36	
TZM 6 D1	3.204	100	320.4	CT&INSRTS			44242	68.19	
TZM 6 D2	0.460	100	46	QW FLTR			8080	12.45	
TZM 6 D3	0.266	100	26.6						
TZM 6 D4	0.147	100	14.7	Total			64882		
TZM 6 D5	0.120	100	12						
TZM 6 D6	0.102	100	10.2						
TZM 6 D7	0.167	100	16.7						
TZM 6 D8	0.425	100	42.5						
TZM 6 D9	39.470	100	3947						
TZM 6 D10	124.700	100	12470						
TZM 6 D11	92.540	100	9254						
TZM 6 D12	63.440	100	6344						
TZM 6 D13	40.130	100	4013						
TZM 6 D14	22.030	100	2203						
TZM 6 D16	3.568	100	356.8						
TZM 6 D17	2.217	100	221.7						
TZM 6 D18	2.251	100	225.1						
TZM 6 D19	1.680	100	168						
TZM 6 D20	1.423	100	142.3						
TZM 6 D21	1.339	100	133.9						
TZM 6 D22	1.379	100	137.9						
TZM 6 D23	1.428	100	142.8						
TZM 6 D24	1.183	100	118.3						
TZM 6 D25	0.948	100	94.8						
TZM 6 D26	1.080	100	108						
TZM 6 D27	0.836	100	83.6						
TZM 6 D28	0.888	100	88.8						
TZM 6 D29	1.430	100	143						
TZM 6 D30	2.259	100	225.9						
TZM 6 D31	2.759	100	275.9						
TZM 6 D32	4.910	100	491						
TZM 6 D33	5.436	100	543.6						
TZM 6 QW	80.800	100	8080						
TZM 6 Col Tube	18.205	100	1820.5						
TZM 6 RXN	125.6	100	12560						
		Total (ug)	64882						
		Total (mg)	64.9						
		Amount excluding RXN and Col tube (mg)	50502

TZM6: Tested at 700°C for 2 hours (500 sccm flow)

	Position	Mass Mo	Mass Mo,ug w Col tube	Mass Mo,mg w Col tube	Temp.,			
	in.	ug	redistributed	redistributed	C			
TZM 6 D1	1.5	320.4	331.9	0.3319				
TZM 6 D2	2.5	46.0	47.7	0.0477		Specimen (cm ²)		
TZM 6 D3	3.5	26.6	27.6	0.0276		9.075		Summer'99
TZM 6 D4	4.5	14.7	15.2	0.0152				
TZM 6 D5	5.5	12.0	12.4	0.0124			16	0
TZM 6 D6	6.5	10.2	10.6	0.0106			17	1
TZM 6 D7	7.5	16.7	17.3	0.0173		Rate (g/m ² -h):	18	2
TZM 6 D8	8.5	42.5	44.0	0.0440		35.75	19	3
TZM 6 D9	9.5	3947.0	4089.3	4.0893			20	4
TZM 6 D10	10.5	12470.0	12919.5	12.9195			21	5
TZM 6 D11	11.5	9254.0	9587.6	9.5876			22	6
TZM 6 D12	12.5	6344.0	6572.7	6.5727			23	7
TZM 6 D13	13.5	4013	4157.7	4.1577			24	8
TZM 6 D14	14.5	2203	2282.4	2.2824			25	9
TZM 6 D16	15.5	356.8	369.7	0.3697			26	10
TZM 6 D17	16.5	221.7	229.7	0.2297			27	11
TZM 6 D18	17.5	225.1	233.2	0.2332			28	12
TZM 6 D19	18.5	168	174.1	0.1741			29	13
TZM 6 D20	19.5	142.3	147.4	0.1474			30	14
TZM 6 D21	20.5	133.9	138.7	0.1387			32	16
TZM 6 D22	21.5	137.9	142.9	0.1429			34	18
TZM 6 D23	22.5	142.8	147.9	0.1479			36	20
TZM 6 D24	23.5	118.3	122.6	0.1226			40	24
TZM 6 D25	24.5	94.8	98.2	0.0982			44	28
TZM 6 D26	25.5	108	111.9	0.1119			48	32
TZM 6 D27	26.5	83.6	86.6	0.0866			49	33
TZM 6 D28	27.5	88.8	92.0	0.0920			50	34
TZM 6 D29	28.5	143	148.2	0.1482			51	35
TZM 6 D30	29.5	225.9	234.0	0.2340			52	36
TZM 6 D31	30.5	275.9	285.8	0.2858				
TZM 6 D32	31.5	491	508.7	0.5087				
TZM 6 D33	32.5	543.6	563.2	0.5632				
TZM 6 QW	34	8080	8371.3	8.3713				
TZM 6 Col Tube		1820.5

TZM12: Tested at 700°C for 2 hours (1000 sccm flow)

[illegible]

TZM9: Tested at 700°C for 2 hours (2500 sccm flow)

Mo Oxidation Test TZM9 ---- 700 for 2 h at 2.5 liters/minute						Mass Mo in		Percent	
						ug	Component		
ICP Results	202.0	336.1	339.1	Volume	Mass Mo				
sample	Mo µg/mL	Ti µg/mL	Zr µg/mL	sample(mL)	ug				
						RXN CHMBR	3442.5	6.07	
						CT&INSRTS	44212.8	77.97	
						QW FLTR	9050	15.96	
TZM 9 H1	15.28	-0.007	0.004	100	1528				
TZM 9 H2	3.031	-0.008	-0.002	100	303.1				
TZM 9 H3	0.683	-0.004	-0.002	100	68.3				
TZM 9 H4	141.400	0.001	0.062	100	14140				
TZM 9 H5	92.780	0.000	0.039	100	9278				
TZM 9 H6	16.260	-0.004	0.005	100	1626				
TZM 9 H7	5.268	-0.004	-0.001	100	526.8				
TZM 9 H8	4.558	-0.007	-0.002	100	455.8				
TZM 9 H9	5.224	-0.006	-0.002	100	522.4				
TZM 9 H10	2.233	-0.006	-0.002	100	223.3				
TZM 9 H11	5.811	-0.005	-0.001	100	581.1				
TZM 9 QW	90.500	0.001	0.039	100	9050				
TZM 9 CT	149.600	0.006	0.068	100	14960				
TZM 9 RXN	13.770	-0.002	0.005	250	3442.5				
			Total (ug)	56705					
			Total (mg)	56.7					
			Amount excluding RXN and Col tube	38302.8					
			Mass Mo (ug)		April,28				
	Position	Mass Mo	w Col tube	Temp. x 10	Temp.,				
	in.	ug	redistributed		C				
TZM 9 H1	2	1528	2124.8	6720	672				
TZM 9 H2	5	303.1	421.5	7060	706				
TZM 9 H3	8	68.3	95.0	6790	679				
TZM 9 H4	11	14140	19662.7	5990	599	Specimen (cm ²)		Summer'99	Temp.(2)
TZM 9 H5	14	9278	12901.7	4750	475	9.069	16	0	652 6520
TZM 9 H6	17	1626	2261.1	3850	385		17	1	668 6680
TZM 9 H7	20	526.8	732.6	3270	327		18	2	675 6750
TZM 9 H8	23	455.8	633.8	2820	282		19	3	683 6830
TZM 9 H9	26	522.4	726.4			Rate (g/m ² -h):	20	4	692 6920
TZM 9 H10	29	223.3	310.5			31.26	21	5	701 7010
TZM 9 H11	32	581.1	808.1				22	6	701 7010
TZM 9 QW	35	9050	12584.7				23	7	692 6920
TZM 9 CT		14960					24	8	682 6820
							25	9	667 6670
							26	10	641 6410
							27	11	601 6010
							28	12	554 5540
							29	13	505 5050
							30	14	470 4700
							32	16	404 4040
							36	20	326 3260
							40	24	250 2500
							44	28	222 2220
							46	30	207 2070
							48	32	172 1720
							49	33	148 1480
							50	34	99 990
							51	35	98 980
							52	36	30 300

TZM3: Tested at 750°C for 1 hour (1000 sccm flow)

These are the analyses associated with Post-test TZM3 (750C/1 h)																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	
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TZM2: Tested at 800°C for 1 hour (1000 sccm flow)

Mo Oxidation Test TZM2 ---- 800C for 1 h at 1.0 liters/minute								Mass Mo	Percent			
ICP Results	202.0	Volume	Mass Mo				ug	in				
sample	Mo µg/mL	Sample(mL)	ug					Component				
					RXN CHMBR		518000	23.35				
TZM 2 E1	6.67	100	667		CT&INSRTS		1604391	72.32				
TZM 2 E2	53.78	100	5378		QW FLTR		95960	4.33				
TZM 2 E3	121.5	100	12150									
TZM 2 E4	13030	100	1303000		Total		2218351					
TZM 2 E5	433.6	100	43360									
TZM 2 E6	522.4	100	52240									
TZM 2 E7	67.48	100	6748									
TZM 2 E8	46.74	100	4674									
TZM 2 E9	55.94	100	5594									
TZM 2 E10	1435	100	143500									
TZM 2 QW	959.6	100	95960									
TZM 2 Col Tube	270.8	100	27080									
TZM 2 RXN	2072	250	518000									
		Total (ug)	2218351									
		Total (mg)	2218.4									
		Amount excluding RXN and Col tube (mg)	1673271									
			Mass Mo,ug	Mass Mo,mg								
	Position	Mass Mo	w Col tube	w Col tube								
	in.	ug	redistributed	redistributed								
TZM 2 E1	2	667.0	677.8	0.6778								
TZM 2 E2	5	5378.0	5465.0	5.4650							July,12	Temp.
TZM 2 E3	8	12150.0	12346.6	12.3466		Specimen (cm ²)					Temp.	x2
TZM 2 E4	11	1303000.0	1324087.6	1324.0876		9.15		16	0	777	1554	
TZM 2 E5	14	43360.0	44061.7	44.0617				17	1	790	1580	
TZM 2 E6	17	52240.0	53085.4	53.0854				18	2	793	1586	
TZM 2 E7	20	6748.0	6857.2	6.8572		Rate (g/m ² -h):		19	3	801	1602	
TZM 2 E8	23	4674.0	4749.6	4.7496		2424.43		20	4	807	1614	
TZM 2 E9	26	5594.0	5684.5	5.6845				21	5	815	1630	
TZM 2 E10	29	143500.0	145822.4	145.8224				22	6	811	1622	
TZM 2 QW	35	95960.0	97513.0	97.5130				23	7	797	1594	
TZM 2 Col Tube		27080.0						24	8	785	1570	
								25	9	770	1540	
								26	10	748	1496	
								27	11	713	1426	
								28	12	666	1332	
								29	13	624	1248	
								30	14	587	1174	
								32	16	522	1044	
								34	18	466	932	
								36	20	417	834	
								40	24	344	688	
								44	28	296	592	
								48	32	175	350	
								49	33	130	260	
								50	34	90	180	
								51	35	72	144	
								52	36	30	60	

Component	Mass Mo (ug)	Percent
RXN CHMBR	518000	23.35
CT&INSRTS	1604391	72.32
QW FLTR	95960	4.33

Distance (in)	Mass (mg)	Temp., Cx(2)
0	0	1554
5	0	1580
10	1324	1602
15	0	1614
20	0	1630
25	0	1594
30	154	1570
35	0	1540